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(株) クレ 知的財産部 倉敷

No. 7185 P. 2

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IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :

YASUO TOKITOH :

EXAMINER: JOHNSON, E.

SERIAL NO: 10/073,225 :

FILED: FEBRUARY 13, 2002 :

GROUP ART UNIT: 1754

FOR: COMPOSITE PRODUCT AND
MANUFACTURING METHOD THEREOF :

DECLARATION UNDER 37 C.F.R. § 1.132

COMMISSIONER FOR PATENTS
ALEXANDRIA, VIRGINIA 22313

SIR:

I, Yasuo Tokitoh, a citizen of Japan, residing at 4-4-65 Seiwadainishi, Kawanishi-shi, Hyogo-ken, Japan do hereby declare as follows:

I am an inventor in the above-identified application.

I graduated from Master Course of Faculty of Chemical Engineering, Tokyo Institute of Technology in March, 1976. I entered Kuraray Co., Ltd. in April 1976, and engaged in the research and development of catalytic reactions and chemical processes.

In order to demonstrate that mixtures of a carrier powder and a metal hydroxide in powder form provide composites having different characteristics when heated at temperatures above 400° in comparison to composites prepared by heating the mixtures of powders at a temperature of between 80 and 400°C, the following experiments were carried out by me or under my direct supervision and control.



Experiment 1: Preparation of composite 1

A 100 ml four-necked separable flask was equipped with a stirrer, a thermometer. Then, 10g of alumina (N611N manufactured by NIKKI Chemical CO., LTD.), and 20g of 95% potassium hydroxide crushed to an average particle size of 1 mm or less were charged therein, and the pressure in the system was reduced to 20 kPa. Heating was started in an oil bath heated to 120°C and the temperature was increased up to 120°C with slow stirring (revolutions per minute 50 rpm). After reaching 120°C, the temperature was increased at 10°C/min, so that the flask was heated up to 450°C. At 450°C, the flask was further heated and dried for another two hours, and then cooled down to room temperature to obtain a composite.

Experiment 2: Preparation of composite 2 (Same as Example 3 of the current Application No. 10/073,225)

In Experiment 1, after increasing the temperature to 120°C, the temperature was increased at 10°C/min, and the flask was heated up to 190°C. At 190°C, the flask was further heated and dried for another two hours, and then cooled down to room temperature to obtain a composite.

Experiment 3: Isomerization reaction of 4-methyl-3,6-dihydro-2H-pyran

Into a 100ml three necked flask, 70g of 4-methyl-3,6-dihydro-2H-pyran and 1.0g of the composite prepared in Experiment 1 were charged. The flask was then equipped with a distillation column packed with a 20-cm Helipack, and with a reflux head and a thermometer, and the atmosphere in the system was replaced with nitrogen. The flask was heated to 120°C, and the distillate solution at an overhead temperature of 98°C was collected. The entire

contents of the flask were distilled for a reaction time of 12 hours to recover 12.1g of 4-methylene-2H-pyran with a purity of 94.7%.

Experiment 4: HCl treatment of composite 1

5.0g of the composite prepared in Experiment 1 were washed with 1000ml of 1N hydrochloric acid, then with 5000ml of ion-exchanged water. The washed composite was then dried at 180°C under the reduced pressure of 130Pa. After drying, the weight of the composite was 3.52g (70.4% of the alumina were recovered).

Experiment 5: HCl treatment of composite 2

5.0g of the composite prepared in Experiment 2 were washed with 1000ml of 1N hydrochloric acid, then with 5000ml of ion-exchanged water. The washed composite was then dried at 180°C under the reduced pressure of 130Pa. After drying, the weight of the composite was 4.805g (96.1% of the alumina were recovered).

Experiment 6: Preparation of composite 3

A 100 ml four-necked separable flask was equipped with a stirrer, a thermometer and a gas inlet tube. Then, 10g of an active carbon (PW manufactured by Kuraray Chemical CO., LTD.), and 10g of 95% potassium hydroxide crushed to an average particle size of 1 mm or less were charged therein, and dry air (21°C, relative humidity 20) was introduced through the inlet tube into the flask at 200 mL/min. Heating was started in an oil bath heated to 120°C and the temperature was increased up to 120°C with slow stirring (revolutions per minute 50 rpm). After reaching 120°C, the temperature was increased at 10°C/min, so that the flask was heated up to 450°C. At 450°C, the flask was further heated and dried for another hour, and then cooled down to room temperature to obtain a composite.

Experiment 7: Preparation of composite 4 (same as Example 2 of current application)

In Experiment 6, after increasing the temperature to 120°C, the temperature was increased at 10°C/min, and the flask was heated up to 180°C. At 180°C, the flask was further heated and dried for another hour, and then cooled down to room temperature to obtain a composite.

Experiment 8: HCl treatment of composite 3

5.0g of the composite prepared in Experiment 6 were washed with 1000mL of 1N hydrochloric acid, then with 5000mL of ion-exchanged water. The washed composite was then dried at 180°C under the reduced pressure of 130Pa. The oxygen content of the obtained composite determined by ECSA was 2.55 wt%.

Experiment 9: HCl treatment of composite 4

5.0g of the composite prepared in Experiment 6 were washed with 1000mL of 1N hydrochloric acid, then with 5000mL of ion-exchanged water. The washed composite was then dried at 180°C under the reduced pressure of 130Pa. The oxygen content of the obtained composite determined by ESCA was 0.96 wt%.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the

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United States Code and that such willful false statements may jeopardize the validity of application of any patent issuing thereon.

Further deponent saith not.

Date: February 17, 2004

Yasuo Tokitoh
Yasuo Tokitoh